

checked by AT
3/3/16

MEMORANDUM

TO: Mr. Addison Rice
Anderson, Mulholland and Associates

DATE: February 25, 2016

FROM: R. Infante 

FILE: 1602064D

RE: Data Validation
Air samples
SDG: 1602064D

SUMMARY

Full validation was performed on the data for several gas samples analyzed for methane by ASTM method D-1946-modified. The samples were collected at the Bristol Myer Squib facility, Humacao, PR site on January 31, 2016 and submitted to Eurofins Air Toxics, Inc. of Folsom, California that analyzed and reported the results under delivery groups (SDG) 1602064D.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006; and the QC criteria of the ASTM method D-1946-modified. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use.

SAMPLES

The samples included in the review are listed below

Client Sample ID	Lab. Sample ID	Collected Date	Matrix	Analysis
B30-1IA013016	1602064D-01A	01/31/2016	Air	Methane
B30-2IA013016	1602064D-02A	01/31/2016	Air	Methane
B30-3IA013016	1602064D-03A	01/31/2016	Air	Methane
B30-4IA013016	1602064D-04A	01/31/2016	Air	Methane
B30-4DIA013016	1602064D-05A	01/31/2016	Air	Methane
B30-5IA01316	1602064D-06A	01/31/2016	Air	Methane
B42-1IA013016	1602064D-07A	01/31/2016	Air	Methane
B42-2IA013016	1602064D-08A	01/31/2016	Air	Methane
B42-3IA013016	1602064D-09A	01/31/2016	Air	Methane
B30-1SSV013116	1602064D-11A	01/31/2016	Air	Methane
B30-2SSV013116	1602064D-12A	01/31/2016	Air	Methane
B30-3SSV013116	1602064D-13A	01/31/2016	Air	Methane
B30-4DSSV013116	1602064D-14A	01/31/2016	Air	Methane
B30-5SSV013116	1602064D-15A	01/31/2016	Air	Methane
B42-1SSV013116	1602064D-16A	01/31/2016	Air	Methane
B42-2SSV013116	1602064D-17A	01/31/2016	Air	Methane
B42-3SSV013116	1602064D-18A	01/31/2016	Air	Methane

REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- Agreement of analysis conducted with chain of custody (COC) form
- Holding time and sample preservation
- Gas chromatography/mass spectrometry (GC/MS) tunes
- Initial and continuing calibrations
- Method blanks/trip blanks/field blank
- Canister cleaning certification criteria
- Surrogate spike recovery
- Internal standard performance and retention times
- Field duplicate results
- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- Quantitation limits and sample results

DISCUSSION

Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody form.

Holding Times and Sample Preservation

Sample preservation was acceptable.

Samples analyzed within method recommended holding time.

Initial and Continuing Calibrations

Methane by ASTM method D-1946 (modified)

Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.

Method Blank/Trip Blank/Field Blank

Target analytes were not detected in laboratory method blanks.

No trip/field blank analyzed with this data package.

Laboratory/Field Duplicate Results

Field/laboratory duplicates were analyzed as part of this data set. Target analytes meet the RPD performance criteria of + 25 % for analytes 5 x SQL.

LCS/LCSD Results

Methane

LCS/LCSD (blank spike) were analyzed by the laboratory associated with this data package. Recoveries and RPD within laboratory control limits.

Quantitation Limits and Sample Results

Dilutions were not performed (see worksheet).

Calculations were spot checked.

Certification

The following samples 1602064D-01A; 1602064D-02A; 1602064D-03A; 1602064D-04A; 1602064D-05A; 1602064D-06A; 1602064D-07A; 1602064D-08A; 1602064D-09A; 1602064D-11A; 1602064D-12A; 1602064D-13A; 1602064D-14A; 1602064D-15A; 1602064D-16A; 1602064D-17A; and 1502113B-18A were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document.


Rafael Infante

Chemist License 1888





Air Toxics

Client Sample ID: B42-1SSV013116

Lab ID#: 1602064D-16A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9021107	Date of Collection: 1/31/16 4:40:00 PM
Dil. Factor:	2.37	Date of Analysis: 2/11/16 12:01 PM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00024	0.000071 J

J = Estimated value.

Container Type: 1 Liter Summa Canister





Air Toxics

Client Sample ID: B42-2SSV013116

Lab ID#: 1602064D-17A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9021108	Date of Collection:	1/31/16 4:06:00 PM
Dil. Factor:	2.26	Date of Analysis:	2/11/16 12:27 PM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00023	0.00025

Container Type: 1 Liter Summa Canister





Air Toxics

Client Sample ID: B42-3SSV013116

Lab ID#: 1602064D-18A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9021109	Date of Collection: 1/31/16 4:24:00 PM
Dil. Factor:	2.27	Date of Analysis: 2/11/16 12:50 PM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00023	0.00020 J

J = Estimated value.

Container Type: 1 Liter Summa Canister





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Sample Transportation Notice

Relinquishing signature on this document indicates that sample is being shipped in compliance with all applicable local, State, Federal, national, and international laws, regulations and ordinances of any kind. Air Toxics Limited assumes no liability with respect to the collection, handling or shipping of these samples. Relinquishing signature also indicates agreement to hold harmless, defend, and indemnify Air Toxics Limited against any claim, demand, or action, of any kind, related to the collection, handling, or shipping of samples. D.O.T. Hotline (800) 467-4922

180 BLUE RAVINE ROAD, SUITE B
FOLSOM, CA 95630-4719
(916) 985-1000 FAX (916) 985-1020

Page 1 of 2

Project Manager Terry Taylor
Collected by: (Print and Sign) Terry Taylor
Company AMAI Email _____
Address 2700 Westchester City Purchase State NY Zip 10591
Phone _____ Fax _____

Project Info:

P.O. # _____

Project # _____

Project Name BMS VI

Turn Around Time:

☒ Normal☐ Rush

specify _____

Lab Use Only

Pressurized by: _____

Date: _____

Pressurization Gas: _____

N₂ He

Lab I.D.	Field Sample I.D. (Location)	Can #	Date of Collection	Time of Collection	Analyses Requested	Canister Pressure/Vacuum			
						Initial (inHg)	Final (inHg)	Receipt	Final (psi)
DIA	B30-1 IA 013016	621261	1-31-16	10:32	Full TO-15, CH ₄ , MeOH	-30+	7.0		
DIA	B30-2 IA 013016	14872	1-31-16	10:33		-30	7.0		
DIA	B30-3 IA 013016	34354	1-31-16	10:42		-30	-7.0		
DIA	B30-4 IA 013016	34212	1-31-16	09:36		-30	-1.5		
DIA	B30-4D IA 013016	N0620	1-31-16	09:36		-26	-10.0		
DIA	B30-5 IA 013016	621296	1-31-16	10:58		-28	-8.0		
DIA	B42-1 IA 013016	34259	1-31-16	11:30		-30+	-8.5		
DIA	B42-2 IA 013016	96107	1-31-16	11:28		-28	-8.0		
DIA	B42-3 IA 013016	N0645	1-31-16	11:29		-30+	-6.0		
DIA	B3042AA 013016	5672	1-31-16	11:07		-30	-8.0		

Relinquished by: (signature) _____ Date/Time _____

Received by: (signature) _____ Date/Time _____

Notes:

Report to MDL

Relinquished by: (signature) _____ Date/Time _____

Received by: (signature) _____ Date/Time _____

Relinquished by: (signature) _____ Date/Time _____

Received by: (signature) _____ Date/Time _____

Lab Use Only	Shipper Name		Air Bill #	Temp (°C)		Condition	Custody Seals Intact?			Work Order #
	Fed Ex			N/A			<input checked="" type="radio"/> Yes	<input type="radio"/> No	<input type="radio"/> None	
										1602064



eurofins

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Sample Transportation Notice

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FOLSOM, CA 95630-4719
(916) 985-1000 FAX (916) 985-1020

Page 2 of 2

Project Manager Terry Taylor
Collected by: (Print and Sign) Terry Taylor
Company AMAI Email _____
Address 2700 Westchester City Purchase State NY Zip 10591
Phone _____ Fax _____

Project Info:

P.O. # _____

Project # _____

Project Name BMS VI

Turn Around Time:

☒ Normal☐ Rush

specify _____

Lab Use Only

Pressurized by:

Date:

Pressurization Gas:

N₂ He

Lab I.D.	Field Sample I.D. (Location)	Can #	Date of Collection	Time of Collection	Analyses Requested	Canister Pressure/Vacuum			
						Initial in Hg	Final in Hg	Receipt	Final (psi)
11A	B30-1SSV013116	1L1536	1-31-16	1224	Full to -15, CH ₄ , MeOH	-30	-5		
12A	B30-2SSV013116	8002	1-31-16	1240		-30	-5		
13A	B30-3SSV013116	34578	1-31-16	1300		-30	-5		
14A	B30-4DSSV013116	1L1942	1-31-16	1320		-25	-5		
15A	B30-5SSV013116	11446	1-31-16	1349		-30	-5		
16A	B42-1SSV013116	34579	1-31-16	1640		-26.5	-4		
17A	B42-2SSV013116	37718	1-31-16	1606		-30	-5		
18A	B42-3SSV013116	36483	1-31-16	1624		-30	-5		

Relinquished by: (signature) [Signature] Date/Time 1/31/16 1725

Received by: (signature) [Signature] Date/Time 1/31/16 1725

Relinquished by: (signature) [Signature] Date/Time 2/1/16 1200

Received by: (signature) [Signature] Date/Time 2/2/16 1020

Relinquished by: (signature) _____ Date/Time _____

Received by: (signature) _____ Date/Time _____

Notes:

Report to MDL

Lab Use Only	Shipper Name	Air Bill #	Temp (°C)	Condition	Custody Seals Intact?	Work Order #
	Epd ex		N/A	Good	<input checked="" type="radio"/> Yes <input type="radio"/> No <input type="radio"/> None	1602064



Air Toxics

Client Sample ID: B30-11A013016

Lab ID#: 1602064D-01A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9021004
Dil. Factor: 1.83

Date of Collection: 1/31/16 10:32:00 AM
Date of Analysis: 2/10/16 09:01 AM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00018	0.00027

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B30-21A013016

Lab ID#: 1602064D-02A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9021005	Date of Collection: 1/31/16 10:33:00 AM
Dil. Factor:	1.71	Date of Analysis: 2/10/16 09:22 AM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00017	0.00027

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B30-31A013016

Lab ID#: 1602064D-03A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9021006	Date of Collection: 1/31/16 10:42:00 AM
Dil. Factor:	1.79	Date of Analysis: 2/10/16 09:48 AM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00018	0.00024

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B30-4IA013016

Lab ID#: 1602064D-04A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9021007	Date of Collection: 1/31/16 9:36:00 AM
Dil. Factor:	1.46	Date of Analysis: 2/10/16 10:12 AM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00015	0.00023

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B30-4DIA013016

Lab ID#: 1602064D-05A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9021008	Date of Collection: 1/31/16 9:36:00 AM
Dil. Factor:	2.01	Date of Analysis: 2/10/16 10:36 AM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00020	0.00023

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B30-51A013016

Lab ID#: 1602064D-06A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9021009
Dil. Factor: 1.87

Date of Collection: 1/31/16 10:58:00 AM
Date of Analysis: 2/10/16 11:18 AM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00019	0.00021

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B42-11A013016

Lab ID#: 1602064D-07A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9021011
Dil. Factor: 1.96

Date of Collection: 1/31/16 11:30:00 AM
Date of Analysis: 2/10/16 12:07 PM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00020	0.00018 J

J = Estimated value.

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B42-2IA013016

Lab ID#: 1602064D-08A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9021012
Dil. Factor: 1.96

Date of Collection: 1/31/16 11:28:00 AM
Date of Analysis: 2/10/16 12:37 PM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00020	0.00021

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B42-3IA013016

Lab ID#: 1602064D-09A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9021013	Date of Collection: 1/31/16 11:29:00 AM
Dil. Factor:	1.71	Date of Analysis: 2/10/16 01:03 PM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00017	0.00019

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B3042AA013016

Lab ID#: 1602064D-10A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9021014
Dil. Factor: 1.91

Date of Collection: 1/31/16 11:07:00 AM
Date of Analysis: 2/10/16 01:48 PM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00019	0.00020

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B30-1SSV013116

Lab ID#: 1602064D-11A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9021110
Dil. Factor: 2.39

Date of Collection: 1/31/16 12:24:00 PM
Date of Analysis: 2/11/16 01:48 PM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00024	48

Container Type: 1 Liter Summa Canister





Air Toxics

Client Sample ID: B30-2SSV013116

Lab ID#: 1602064D-12A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9021015	Date of Collection:	1/31/16 12:40:00 PM
Dil. Factor:	2.32	Date of Analysis:	2/10/16 02:13 PM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00023	0.00029

Container Type: 1 Liter Summa Canister





Air Toxics

Client Sample ID: B30-3SSV013116

Lab ID#: 1602064D-13A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9021016	Date of Collection:	1/31/16 1:00:00 PM
Dil. Factor:	2.51	Date of Analysis:	2/10/16 02:42 PM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00025	0.00066

Container Type: 1 Liter Summa Canister





Air Toxics

Client Sample ID: B30-4DSSV013116

Lab ID#: 1602064D-14A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9021017	Date of Collection:	1/31/16 1:20:00 PM
Dil. Factor:	2.56	Date of Analysis:	2/10/16 03:08 PM

Compound	Min. Det. Limit (%)	Amount (%)
Methane	0.00026	0.00018 J

J = Estimated value.

Container Type: 1 Liter Summa Canister



DATA REVIEW WORKSHEETS

Project Number: 1602064D

Date: 01/30/2016

REVIEW OF VOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: QC criteria from ASTM D-1946 method for measuring permanent gases and light hydrocarbons in refinery and other sources samples using gas chromatography (GC) and a thermal conductivity detector (TCD) and/or flame ionization detection (FID). Validating Air Samples. Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006). The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Eurofins data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: 1602064D

Sample matrix: Air

No. of Samples: 18

Trip blank No.: -

Field blank No.: -

Equipment blank No.: -

Field duplicate No.: B30-4IA013016/B30-4DIA013016

☒ Data Completeness

☒ Holding Times

☐ N/A GC/MS Tuning

☐ N/A Internal Standard Performance

☒ Blanks

☐ N/A Surrogate Recoveries

☐ N/A Matrix Spike/Matrix Spike Duplicate

☒ Laboratory Control Spikes

☒ Field Duplicates

☒ Calibrations

☒ Compound Identifications

☒ Compound Quantitation

☒ Quantitation Limits

Overall Comments: Methane_by_ASTM_method_D-1946_(modified)

Definition of Qualifiers:

J- Estimated results

U- Compound not detected

R- Rejected data

UJ- Estimated nondetect

Reviewer: Rafael Defant

Date: 02/26/2016

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples analyzed within the recommended method holding time				

Criteria

Aqueous samples – 14 days from sample collection for preserved samples ($\text{pH} \leq 2$, 4°C), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C , no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: $4 \pm 2^{\circ}\text{C}$): N/A – summa canisters

Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solid of soil samples is $< 10\%$, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted ($> 10^{\circ}\text{C}$), estimate positive results (J) and nondetects (UJ).

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 05/15/15
 Dates of continuing calibration: 02/10/16
 Instrument ID numbers: GC-9
 Matrix/Level: Air/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.					

Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.
 All %RSD must be $\leq 15\%$ regardless of method requirements for CCC.
 All %Ds must be $\leq 30\%$ regardless of method requirements for CCC.
 Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of ≥ 0.995 has therefore been utilized as professional judgment.

Actions

If any compound has an initial RF or a continuing RF of < 0.05 , estimate positive results (J) and reject nondetects (R), regardless of method requirements.
 If any compound has a %RSD $> 15\%$, estimate positive results (J) and use professional judgment to qualify nondetects.
 If any compound has a %RSD $> 90\%$, estimate positive results (J) and reject nondetects (R).
 If any compound has a % D $> 30\%$, estimate positive results (J) and reject nondetects (R).
 If any compound has a % D $> 30\%$, estimate positive results (J) and nondetects (UJ).
 If any compound has a % D $> 90\%$, estimate positive results (J) and reject nondetects (R).
 If any compound has $r < 0.995$, estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

All criteria were met X
Criteria were not met
and/or see below _____

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
All_method_blank_meeth_method_specific_criteria				

Field/Equipment/Trip blank

[illegible]

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

V B. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and \leq AL, report the compound as not detected (U) at the SQL.

If the concentration is \geq SQL but \leq AL, report the compound as not detected (U) at the reported concentration.

If the concentration is \geq SQL and $>$ AL, report the concentration unqualified.

Notes:

High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES

DATA REVIEW WORKSHEETS

All criteria were met N/A
 Criteria were not met
 and/or see below

SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND	ACTION
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 Surrogate standards not required by the method.

QC Limits* (Air)

 LL to UL to to to

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 80 – 120 % for aqueous and 70 – 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

DATA REVIEW WORKSHEETS

All criteria were met _____
 Criteria were not met _____
 and/or see below __N/A__

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: _____ - _____ Matrix/Level: _____ - _____

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
_MS/MSD_are_not_required_as_part_of_ASTM-method_D-1946;_blank_spike_used_to_assess_ _accuracy_					

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (JJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

DATA REVIEW WORKSHEETS

All criteria were met _____
Criteria were not met _____
and/or see below N/A

VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD – Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID: _____ Matrix/Level/Unit: _____

COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION
----------	-----------------	----------	-----------	-------	--------

[illegible]

Actions:

- * If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).
* If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD?
 Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT
____LCS/LCSD_(Blank_spike)_analyzed_in_this_data_package;_recoveries_and_RPD____			
____within_laboratory_control_limits.____			

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

IX. FIELD/LABORATORY DUPLICATE PRECISION

Sample ID_ B30-4IA013016/B30-4DIA013016_____

Matrix: Air

Field/laboratory duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD \pm 25% for air samples. If both sample and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
RPD for field duplicates within laboratory control limits. RPD for laboratory duplicate (LCS/LCSD) within laboratory control limits.					

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

DATA REVIEW WORKSHEETS

All criteria were met NA
 Criteria were not met
 and/or see below _____

X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- * Area of +40% or -40% of the IS area in the associated calibration standard.
- * Retention time (RT) within ± 0.06 seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
------	-----------	--------	---------	------------------	--------

Internal standard not required by the method. Samples quantified by external standard
method

Actions:

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%		IS AREA > +40%
Positive results	J		J
Nondetected results	R		ACCEPT

2. If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

1510223C-11A

Methane RF = 157692659

$$[] = (245522)/(157692659)$$

$$= 0.000156 \% \text{ OK}$$

All criteria were met X
Criteria were not met
and/or see below _____

A. Dilution performed

[illegible]

List samples which have $\leq 50\%$ solids

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R)